Docket No. 215472US0

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

Klaus Schultes, et al. : GROUP ART UNIT: 1714

SERIAL NO: 10/029,289 :

FILED: DECEMBER 28, 2001 : EXAMINER: SZEKELY, P.A.

FOR: PROCESS FOR PREPARING BEAD POLYMERS WITH AN AVERAGE PARTICLE SIZE IN THE RANGE FROM 1 TO 40 μM, MOULDING COMPOSITIONS COMPRISING BEAD POLYMER, AND MOULDINGS AND PAMA PLASTISOLS

DECLARATION UNDER 37 C.F.R. §1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VA 22313-1450

SIR:

Now comes Dr. Sabine Schwarz-Barać, who deposes and states that:

1. That I received my doctor's degree in polymer chemistry from the Heinrich-Heine-University of Düsseldorf in the year 2003.

2. That I have been employed by Röhm GmbH & Co. KG, for seven months as chemist in the field of research and development.

3. That the following analysis was carried out by me or under my direct supervision and control.

4. That the present invention provides, in part, a mixture prepared by a process for preparing bead polymers having an average particle size of 1 to 40 μm, comprising:

contacting:

at least one polymerizable mix which comprises at least 50% by weight of at least one (meth)acrylate monomer,

at least one aluminum compound, and

an aqueous phase,

to prepare a mixture;

dispersing said mixture at a shear rate $\geq 10^3\ s^{-1}$ to form a dispersion, wherein said dispersion is stabilized by said aluminum compound; and

polymerizing to produce bead polymers having an average particle size of 1 to 40 μm (Claim 16).

5. That the process of the present invention imparts surprisingly superior properties upon the mixture obtained thereby and the products containing the same. Specifically, the process of the present invention surprisingly permits the recovery of bead polymers that exhibit a narrow size distribution and a smaller particle size as compared to that which is conventionally obtainable.

As shown by the data that follows, these results are provided by the step: "dispersing said mixture at a shear rate $\geq 10^3\ s^{-1}$ to form a dispersion, wherein said dispersion is stabilized by said aluminum compound." More particularly, the data below show that the bead polymers having an average particle size within the range of 5-40 μm possess the aforementioned unexpected properties when dispersing the mixture at a shear rate $\geq 10^3\ s^{-1}$ as compared to a shear rate that is below this claimed range.

6. That specimen pieces with dimensions of 60 x 45 x 3 mm were produced as set forth in Example 1 of the present invention and subjected to measurements. Like specimen pieces were obtained by an analogous procedure and, in this evaluation, the only variable was the shear rate. The resultant bead polymers were analyzed for the particle-size distribution together with standard deviation, transmission, yellowness index, and energy half-value angle. The results are shown in the table below:

	Shear	Shear rate	Stirring	Median value of particle-size distribution per Galai CIS	Standard deviation per Galai CIS	Transmission [%]	Yellowness index	Energy half-value angle β [°]
Example 1 of the present invention	7000 rpm for 15 min	1750 s^{-1}	600 rpm	7.1 μm	2.63 μm	76.3	9.4	22.5
Comparative Example 15453/70	3400 rpm for 15 min	850 s^{-1}	600 rpm	17.6 μm	5.3 μm	87.2	5.16	18.7
Comparative Example 15453/72	without shear	0 s^{-1}	600 rpm	36.2 μm	12.0 μm	89.8	3.19	16.6

7. Brief discussion of the results obtained for Comparative Example 15453/70:

With shear at 3400 rpm, distinctly broader distribution; ReMo F3: 0.01%, F67 0.01%

Visual evaluation of Comparative Example 15453/70 (shear at 3400 rpm):

The particles are polydisperse and have a relatively broad particle-size distribution. The main fraction is in the range of about 10 to 25 µm. Under the microscope, larger particles up to about 50 µm and smaller particles (about 1 µm) are observed. The particle structure is mainly round and smooth, and only some relatively large beads are oval. The distribution is distinctly broader than that of product sheared at 7000 rpm.

Particle-size distribution per Mastersizer: 16.4 µm, standard deviation: 8.3 µm

Median value per Galai CIS: 17.6 µm, standard deviation: 5.3 µm

Remo F3: 0.01%, F67: 0.01%

<u>Optical data</u>:

Transmission:	87.2%
Intensity half-value angle:	21.4 °
Scattering coefficient:	0.3
Yellowness index:	5.16

8. Brief discussion of the results obtained for Comparative Example 15453/72:

Without shear, very broad particle-size distribution; main fraction 10 to 100 µm; smaller particles (around 1 µm) are also observed; ReMo F3: 0.01%, F67 0.01%

Visual evaluation of Comparative Example 15453/72: no shear, stirring 600 rpm:

The particles are polydisperse and have a very broad particle-size distribution. Main fraction in the range of about 10 to 100 µm. Smaller particles (about 1 µm) are observed. The particle structure is mainly round and smooth.

Particle-size distribution per Mastersizer: 37.0 µm, standard deviation: 20.8 µm

Median value per Galai CIS: 36.2 µm, standard deviation: 12.0 µm

Remo F3: 0.01%, F67: 0.01%

Optical data:

Transmission:	89.8%
Intensity half-value angle:	16.3 °
Scattering coefficient:	0.22
Yellowness index:	3.19

9. Comparison with standard 1011F beads (standard synthesis):

Comparison values LJ14823/30, 6% in 7N (shearing of the beads at 7000 rpm)

Particle-size distribution - Median value: 18.6 µm, standard deviation: 7.25 µm

Optical data:

Transmission:	89.7%
Intensity half-value angle:	22.9 °
Yellowness index:	3.7

10. That from the data above, it is clear that the claimed process provides beads that have a narrow size distribution and a smaller overall particle size. These test results show that the scattering beads prepared by the process of the present invention and compounded

into moulding compositions (Example 1 of the present invention) scatter the light very effectively without substantial energy loss (i.e., the energy half-value angle is much poorer when the shear rate is less than $\geq 10^3\ s^{-1}$).

11. I declare further that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

12. Further Declarant saith not.

Sabine Schwarz-Barać — 2004-02-09

Name: Sabine Schwarz-Barać — Date: